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D2-'META'-CARBORANE-SILOXANES. III. PREPARATION AND PROPERTIES --ETC(U)  
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D2-'META'-CARBORANE-SILOXANES

III. PREPARATION AND PROPERTIES  
OF TRIFLUOROPROPYL MODIFIED POLYMERS

UNION CARBIDE CORPORATION,  
TARRYTOWN, NEW YORK

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D<sub>2</sub>-meta-Carborane-Siloxanes. III.

Preparation and Properties of  
Trifluoropropyl Modified Polymers

by

E. N. Peters, D. D. Stewart, J. J. Bohan  
R. Moffitt, C. D. Beard, G. T. Kwiatkowski, and E. Hedaya

February 2, 1976



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# ABSTRACT

A series of linear, high molecular weight, trifluoropropyl substituted D<sub>2</sub>-m-carborane-siloxanes were prepared. These polymers were not as stable as conventional D<sub>2</sub>-m-carborane-siloxanes, however they exhibited greater thermal and oxidative stability than fluorosilicones. Indeed from TGA studies, the fluorosilicones start to lose weight 50 to 100° below the onset of weight loss for the trifluoropropyl substituted carborane-siloxnes.

Improved solvent resistance was obtained by the incorporation of trifluoropropyl groups onto the carborane-siloxane backbone. The tris-trifluoropropyl substituted polymer exhibited low swelling in hydrocarbon based solvents and fuels which was similar to results with fluorosilicones.

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D<sub>2</sub>-meta-CARBORANE-SILOXANES. III.  
PREPARATION AND PROPERTIES OF  
TRIFLUOROPROPYL MODIFIED POLYMERS

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Synopsis

Linear D<sub>2</sub>-meta carborane-siloxanes with one, two, and three trifluoropropyl moieties per repeat unit were prepared by the condensation reaction between bis-ureidosilanes and carborane disilanol. Molecular weights between 100,000 and 220,000 were obtained. Compared to fluorosilicones, the trifluoropropyl modified carborane-siloxanes exhibited greater thermal and oxidative stability and in certain cases comparable solvent resistance. The swelling index for carborane-siloxane elastomers decreased with increasing trifluoropropyl content, although this fluorocarbon moiety did compromise the thermal stability of the carborane system.

INTRODUCTION

D<sub>2</sub>-meta-decacarborane-siloxanes are noted for their outstanding thermal stability and flame resistance.<sup>1</sup> Car-

borane systems have been produced with useful elastomeric properties and good strength retention after heat aging in air at 315°C for 300 hours.<sup>1,2</sup> However, the carborane-siloxane elastomers exhibit large swelling in hydrocarbon-based solvents and fuels, similar to that experienced with conventional silicone elastomers.

The role of fluorine and the carbon-fluorine bond in achieving a high degree of solvent resistance and stability is well known in organic polymers.<sup>3</sup> Highly fluorinated polymers are stable and have remarkable resistance to oxidative attack, flame, chemicals, and solvents. These beneficial effects have been attributed to the strength of the carbon-fluorine bond compared to that of the carbon-carbon bond, to steric hindrance, and to strong Van der Waals forces.<sup>4,5</sup>

In fluorine-containing silicones, the position of the fluorine on the alkyl side chain influences the thermal and hydrolytic stability of the compound.<sup>6</sup> Fluorine in the  $\alpha$  or  $\beta$  position undergo hydrolytic or thermal cleavage. Thus the  $\gamma$  position is preferred for maximum stability.<sup>6</sup> For example, poly( $\gamma, \gamma', \gamma''$ -trifluoropropylmethyl)siloxane has excellent solvent resistance. It is reported to be superior in many respects to the fluorocarbon elastomers, but fall short of them at temperatures about 230°C.<sup>6,7</sup> Fluorosilicone as well as conventional dimethylsilicone polymers undergo depolymerization (reversion) at high temperatures, with resultant deterioration of physical properties.

The incorporation of the meta-decacarborane moiety into the siloxane backbone significantly enhanced the overall thermal stability and retarded depolymerization.<sup>8,9</sup> Therefore, trifluoropropyl substituted carborane-siloxanes should similarly exhibit better solvent resistance than carborane-siloxanes and enhanced thermal stability over fluorosilicones. Trifluoropropyl substituted Dexsil 300 (i.e., D<sub>3</sub>-meta-carborane-siloxane) has been reported to be more thermally stable than fluorinated silicones with comparable resistance to solvents.<sup>10</sup> However, the D<sub>3</sub>-polymers do not possess the high-temperature performance potential of the D<sub>2</sub>-polymers.<sup>11</sup> The preparation and characterization of high molecular weight trifluoropropyl modified D<sub>2</sub>-polymers has not been previously described although low molecular weight trifluoropropyl substituted D<sub>2</sub>-carborane-siloxanes have been reported.<sup>12,13</sup>

Recently, we reported a new facile route to linear, high molecular weight D<sub>2</sub>-polymers. We have applied this technique to the preparation of high molecular weight trifluoropropyl substituted D<sub>2</sub>-m-carborane-siloxanes. The synthesis and properties of these polymers are reported herein.

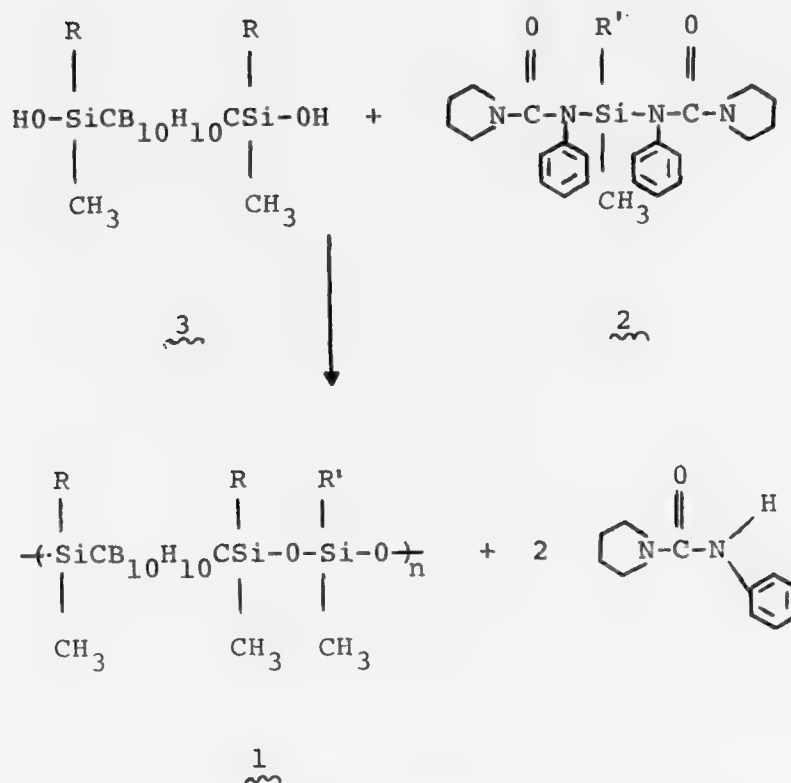
#### RESULTS

Serious fabrication and vulcanization difficulties have prevented exploitation of previously reported carborane-siloxanes such as Dexsil 200. These difficulties were relat-

ed to the mode of polymer synthesis, which involved a ferric chloride catalyzed condensation of a chlorosilane with an alkoxysilane.<sup>14</sup> The ferric chloride induced a crosslinking process during the reaction to give a low molecular weight network polymer, rather than a truly linear product.<sup>9,15</sup> In order to obviate the deleterious effects of ferric chloride, a new facile route was developed based on the condensation of carborane disilanol with a reactive silane comonomer.<sup>1</sup> This condensation reaction provides high molecular weight, linear polymers in which structural modifications can be readily achieved. These unique polymers were given the designation of UCARSIL polymers.

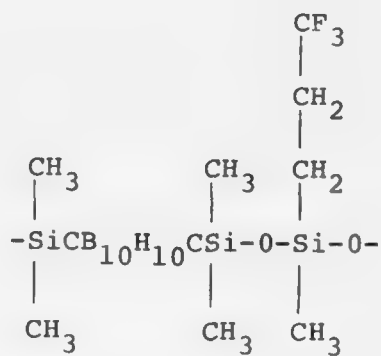
#### Synthesis

The improved polymeriazation process was employed in the preparation of high molecular weight trifluoropropyl modified D<sub>2</sub>-m-carborane-siloxanes, 1, by the condensation of bis (N-pyrrolidino-N'-phenylureido)dialkylsilanes, 2, with bis (hydroxydialkyl-silyl)-meta-carborane, 3, (where the dialkyl groups are either methyltrifluoropropyl or dimethyl). Compound 3 is added slowly to a chlorobenzene solution of 2 at 0 to -10°C.

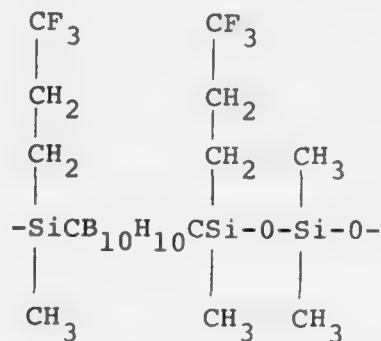


Nuclear magnetic resonance spectroscopy was used to monitor the reaction stoichiometry, permitting adjustments for side reactions and monomer impurities. This technique will be described in detail elsewhere.

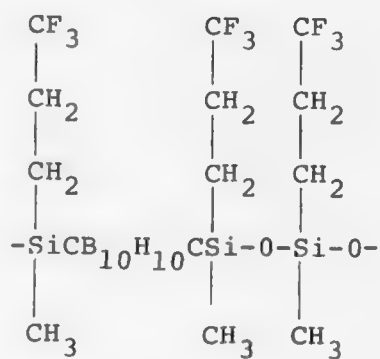
Thus polymers containing one, two, and three methylfluoropropyl groups per repeat unit were prepared and will be designated UCARSIL F<sub>1</sub>, UCARSIL F<sub>2</sub>, and UCARSIL F<sub>3</sub> polymers respectively. These polymers also contained 4 mole % methylvinylsiloxane moieties to facilitate vulcanization.



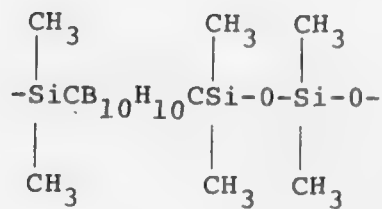
UCARSIL F<sub>1</sub>



UCARSIL F<sub>2</sub>



UCARSIL F<sub>3</sub>



UCARSIL Me<sub>2</sub>

### Polymer Characterization

The trifluoropropyl substituted carborane-siloxanes and D<sub>2</sub>-m-carborane-dimethylsiloxane, UCARSIL Me<sub>2</sub> polymer, were characterized by differential scanning calorimetry (DSC). Over the scanned temperature range (-53° to 200°C), the trifluoropropyl modified polymers were amorphous whereas UCARSIL Me<sub>2</sub> polymer was crystalline (m.p. ~80°C). The glass transition temperatures (T<sub>g</sub>) and molecular weights (via GPC) are listed in Table I.

The glass transtion temperature increased with the number of methyltrifluoropropyl groups. The -CH<sub>2</sub>CH<sub>2</sub>CF<sub>3</sub> group is bulkier than a -CH<sub>3</sub> group; therefore, as the methyl groups are replaced with -CH<sub>2</sub>CH<sub>2</sub>CF<sub>3</sub> the polymer chain mobility was decreased and the T<sub>g</sub> increased. This is consistent with literature data which report the T<sub>g</sub> of poly(methyltrifluoropropylsiloxane) as -75°C versus -123°C for poly(dimethylsiloxane).<sup>16</sup>

### Thermal Stability

The thermal stabilities of UCARSIL polymers and polymethyltrifluoropropylsiloxane were determined by thermal gravimetric analysis (TGA) in air and nitrogen as shown in Figures 1 and 2.

The rate of weight loss and the overall weight loss increased with the amount of trifluoropropyl groups. The fluorosilicone was the least stable polymer and the UCARSIL Me<sub>2</sub> polymer is the most stable. In nitrogen the trifluoropropyl substituted UCARSIL polymers underwent initial weight loss at comparable temperatures (350 - 390°C). However, the onset of weight loss for the fluorosilicone occurred at 275°C.

The TGA of these polymers in air showed a behavior similar to that observed in nitrogen, however the overall weight losses are much less, presumably because of oxidative crosslinking. On the other hand, the weight loss of the fluorosilicone was more rapid in air than in nitrogen. Clearly the incorporation of the m-carborane moiety into the methyltrifluoropropylsiloxane backbone increased the thermal and oxidative stabilities.

The percent weight loss of the thermograms of the trifluoropropyl modified UCARSIL polymers is suggestive that the polymers are degrading in part with the loss of HF at 300 to 450°C followed by the loss of a C<sub>2</sub>F<sub>2</sub> species at temperatures above 450°C.

### Elastomer Properties

Mechanical properties and solvent resistance for vulcanizates of UCARSIL samples were studied. The vulcanizates were prepared by compounding 30 phr reinforcing filler, 2.5 phr ferric oxide (an antioxidant), and 2.5 phr dicumyl peroxide into the UCARSIL stock. This material was then cured.

Heat aging studies on diphenylsiloxane modified UCARSIL vulcanizate showed that the retention of mechanical properties was dependent on the surface characteristics of the filler.<sup>1, 2</sup> A fumed silica, Cab-O-Sil, gave poor retention of properties after heat aging presumably because of silanol-phenylsilane condensation reactions which increased the cross-link density.<sup>1, 2</sup> However, use of Tullanox 500, a fumed silica, which had been trimethylsilylated, resulted in good retention of mechanical properties after heat aging in air at 315°C for periods up to 300 hours (Table II).

The effect of filler on UCARSIL F<sub>1</sub> polymer was evaluated. Mechanical properties as a function of temperature for UCARSIL F<sub>1</sub> vulcanizates containing Cab-O-Sil and Tullanox 500 are shown in Table III. The Cab-O-Sil was a better reinforcing agent. At low temperatures the properties change markedly in Table III due to the glass transition temperature of -29°C.

The poorer thermal stability of UCARSIL F<sub>1</sub> polymer compared to conventional UCARSIL polymers is reflected in the rapid change in mechanical properties after heat aging in air

at 315°C after 25 hours (Table IV versus Table II).

Heat aging in air at 290°C showed a substantial improvement in the retention of properties. Mechanical properties at 25°C and 290°C before and after heat aging up to 150 hours are listed in Table V. Interestingly, Cab-O-Sil did not have any deleterious effects compared to Tullanox 500 on the aging properties as was noted with the diphenyl modified UCARSIL polymer. This corroborates the proposal that phenylsilane-silanol crosslinking reactions are responsible in part for the loss of properties. Since Cab-O-Sil was a better reinforcing filler its use was emphasized with UCARSIL F<sub>2</sub> and F<sub>3</sub> polymers.

Resistance to various solvents is an important characteristic of high performance elastomers. UCARSIL F<sub>1</sub> vulcanizate was immersed for seven days in various solvents at ambient temperatures (ASTM test method D471-72) and showed only minor changes in mechanical properties (Table VI). However, large volume increases in certain solvents was observed. The percent swelling for UCARSIL F<sub>1</sub> vulcanizate with Cab-O-Sil and Tullanox 500 are compared in Table VII with previously reported data for methylphenylsiloxane modified UCARSIL vulcanizate reinforced with Tullanox 500. UCARSIL F<sub>1</sub> vulcanizate exhibited a slightly lower swelling than the conventional UCARSIL material. This swelling was further decreased by the use of Cab-O-Sil as a filler compared to Tullanox 500.

Additional improvements in swelling were desired. Hence, compounded and cured UCARSIL  $F_3$  and  $F_2$  polymers were evaluated. Mechanical properties of UCARSIL  $F_3$  vulcanizate as a function of temperature are listed in Table VIII. The low temperature properties are limited by the high glass transition temperature. Heat aging UCARSIL  $F_2$  and  $F_3$  vulcanizates in air at  $290^\circ$  showed comparable stability with UCARSIL  $F_1$  vulcanizate (Table IX). The percent swelling of the trifluoropropyl modified UCARSIL polymers in certain solvents are compared in Table X. Thus there is a substantial decrease in swelling as the concentration of trifluoropropyl groups increases. Moreover, UCARSIL  $F_3$  vulcanizate has a comparable solvent resistance to the fluorosilicones.

#### CONCLUSIONS

A series of linear, high molecular weight, trifluoropropyl substituted  $D_2$ -m-carborane-siloxanes were prepared. These polymers were not as stable as conventional  $D_2$ -m-carborane-siloxanes, however they exhibited greater thermal and oxidative stability than fluorosilicones. Indeed from TGA studies, the fluorosilicones start to lose weight 50 to  $100^\circ$  below the onset of weight loss for the trifluoropropyl substituted carborane-siloxanes.

Improved solvent resistance was obtained by the incorporation of trifluoropropyl groups onto the carborane-siloxane backbone. The tris-trifluoropropyl substituted

polymer exhibited low swelling in hydrocarbon based solvents and fuels which was similar to results with fluorosilicones.

The authors are grateful for support in part by the Office of Naval Research. We also wish to thank Drs. A. B. Furtek and B. L. Joesten for the TGA and DSC analysis.

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TGA in air of fluorosilicone and UCARSIL  
polymers (heating rate 10°C/min.)

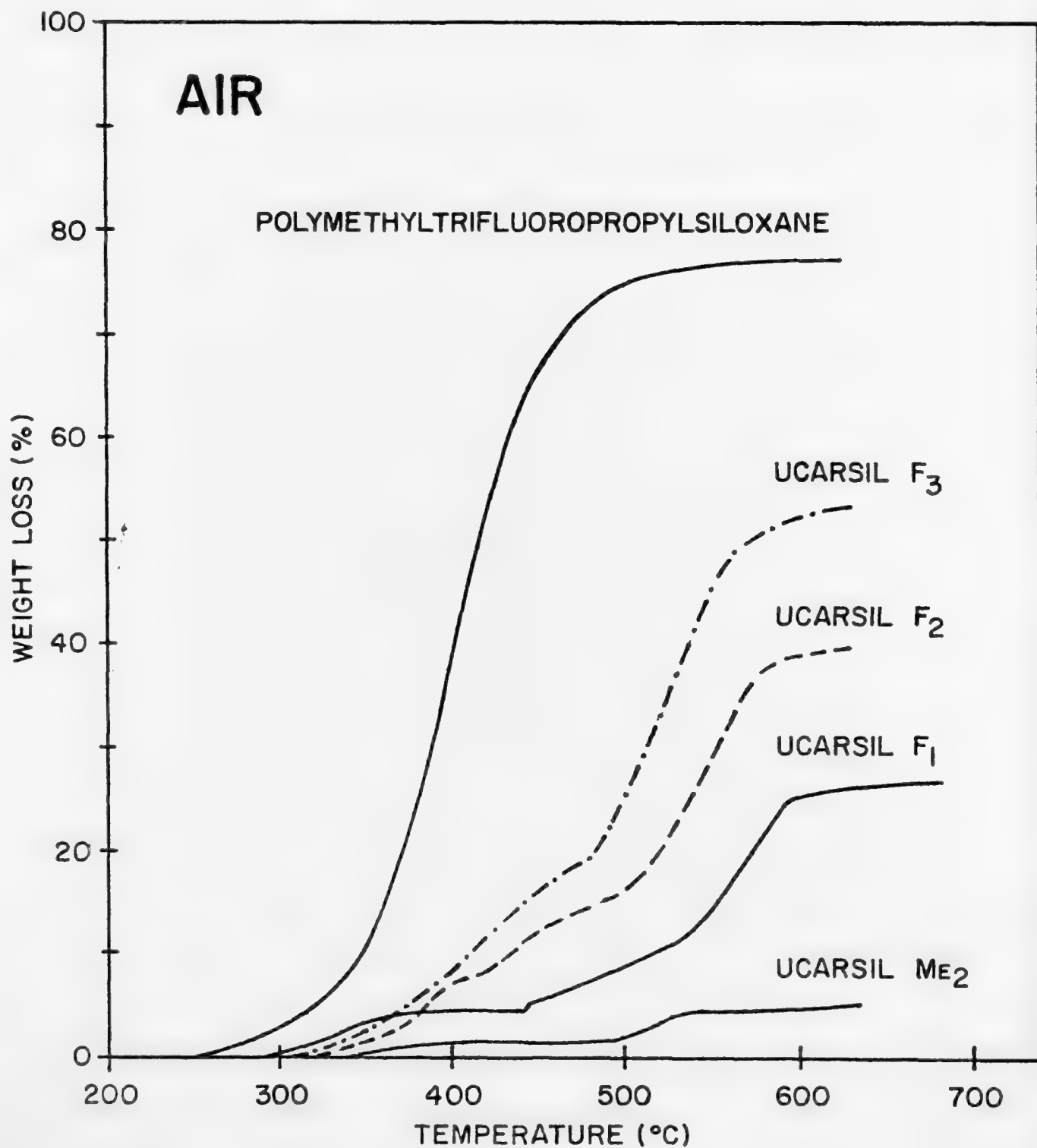


FIGURE 2.

TGA in nitrogen of fluorosilicone and  
UCARSIL polymers (heating rate 10°C/min.)

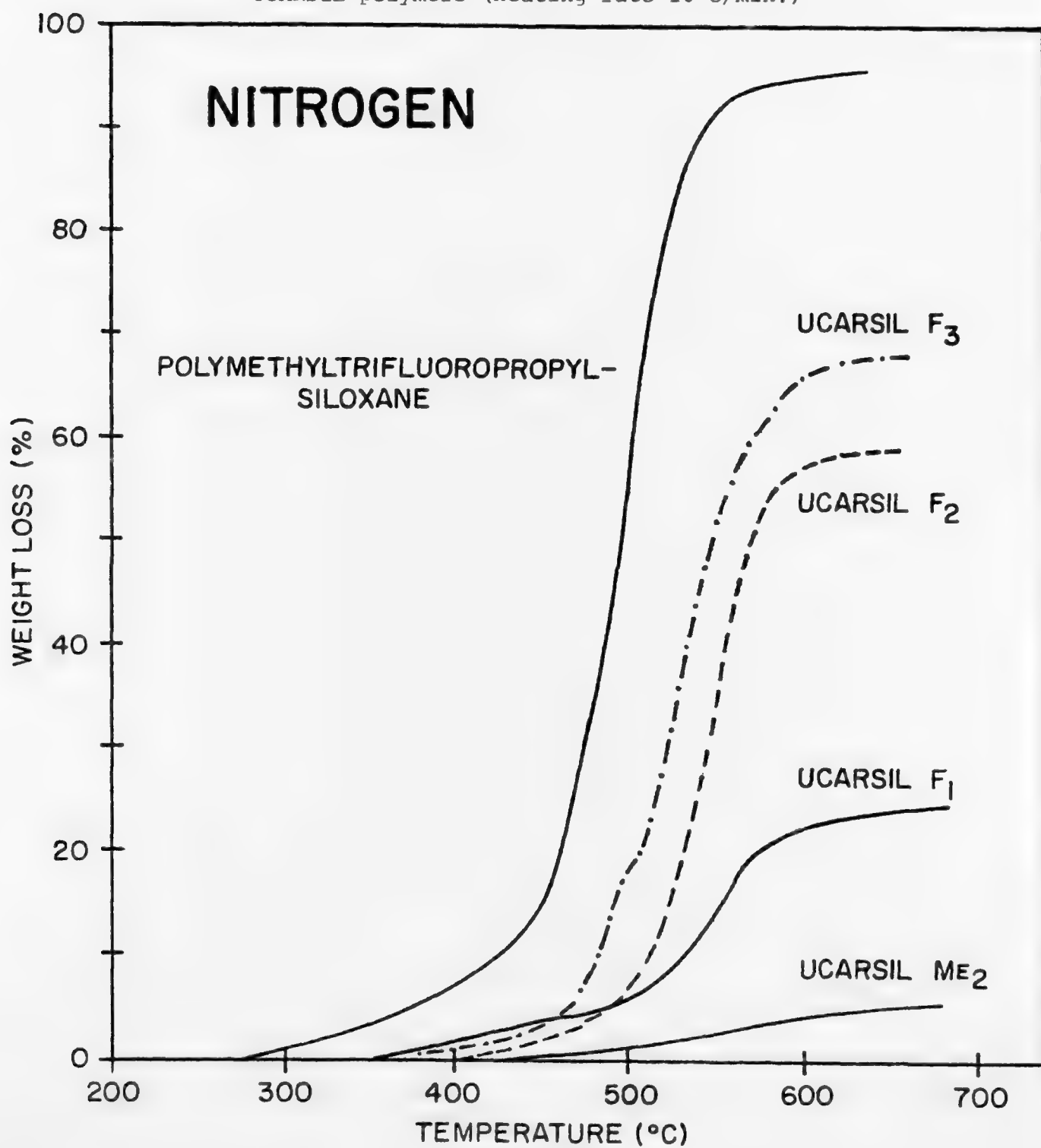


TABLE I

Glass Transition Temperatures  
of D<sub>2</sub>-m-Carborane-Siloxanes<sup>a</sup>

| Polymer                 | CH <sub>2</sub> CH <sub>2</sub> CF <sub>3</sub><br>Groups | $\bar{M}_w$ | T <sub>g</sub><br>°C |
|-------------------------|-----------------------------------------------------------|-------------|----------------------|
| UCARSIL Me <sub>2</sub> | 0                                                         | 110,000     | -50                  |
| UCARSIL F <sub>1</sub>  | 1                                                         | 220,000     | -29                  |
| UCARSIL F <sub>2</sub>  | 2                                                         | 102,000     | -12                  |
| UCARSIL F <sub>3</sub>  | 3                                                         | 149,000     | -3                   |

<sup>a</sup> Determined by DSC (at 10° per minute)

TABLE II

HEAT AGING STUDIES (315°C IN AIR):  
DIPHENYL MODIFIED UCARSIL ELASTOMER

| Aging<br>Time,<br>Hrs.                | Young's<br>Modulus,<br>psi | Tensile<br>Strength,<br>psi | Elongation<br>at Break,<br>% |
|---------------------------------------|----------------------------|-----------------------------|------------------------------|
| <hr/> Tullanox 500 <sup>a</sup> <hr/> |                            |                             |                              |
| 0                                     | 533                        | 613                         | 100                          |
| 25                                    | 922                        | 442                         | 60                           |
| 50                                    | 1117                       | 427                         | 55                           |
| 150                                   | 1813                       | 533                         | 45                           |
| 300                                   | 3400                       | 570                         | 30                           |
| <hr/> Cab-0-Sil <hr/>                 |                            |                             |                              |
| 0                                     | 2000                       | 907                         | 130                          |
| 25                                    | 2180                       | 578                         | 60                           |
| 50                                    | 3370                       | 539                         | 30                           |
| 150                                   | 19400                      | 1129                        | 10                           |
| 300                                   | b                          | b                           | b                            |

<sup>a</sup>Data from reference 2

<sup>b</sup>Too brittle to measure

TABLE III  
Mechanical Properties as a Function of Temperature: UCARSIL F<sub>1</sub> Elastomer

| Temp.,<br>°C | Tullanox 500               |                             |                              | Cab-0-Sil                  |                             |                              |
|--------------|----------------------------|-----------------------------|------------------------------|----------------------------|-----------------------------|------------------------------|
|              | Young's<br>Modulus,<br>psi | Tensile<br>Strength,<br>psi | Elongation<br>at Break,<br>% | Young's<br>Modulus,<br>psi | Tensile<br>Strength,<br>psi | Elongation<br>at Break,<br>% |
| 290          | 408                        | 52                          | 15                           | 1,230                      | 160                         | 20                           |
| 260          | 420                        | 92                          | 30                           | 1,130                      | 198                         | 20                           |
| 200          | 489                        | 149                         | 40                           | 1,350                      | 205                         | 30                           |
| 25           | 783                        | 540                         | 100                          | 1,120                      | 640                         | 110                          |
| -20          | 21,400                     | 1,130                       | 15                           | 22,200                     | 1,250                       | 15                           |
| -30          | 221,000                    | 2,470                       | 2                            | 115,000                    | 2,970                       | 3                            |

TABLE IV

Mechanical Properties at 25°C  
After Heat Aging in Air at 315°C

| Aging<br>Time,<br>HRS. | Young's<br>Modulus,<br>psi | Tensile<br>Strength,<br>psi | Elongation<br>at Break,<br>% |
|------------------------|----------------------------|-----------------------------|------------------------------|
| 0                      | 783                        | 540                         | 100                          |
| 25                     | 7,400                      | 200                         | 2                            |
| 50                     | a                          | a                           | a                            |

a) Too brittle to measure

TABLE V

Heat Aging Studies (290°C in Air) : UCARSIL F<sub>1</sub> Elastomer

| Aging<br>Time,<br>Hrs.   | Young's<br>Modulus,<br>psi |      | Tensile<br>Strength,<br>psi |      | Elongation<br>at Break,<br>% |      |
|--------------------------|----------------------------|------|-----------------------------|------|------------------------------|------|
|                          | 25°                        | 290° | 25°                         | 290° | 25°                          | 290° |
| <hr/> Tullanox 500 <hr/> |                            |      |                             |      |                              |      |
| 0                        | 783                        | 408  | 540                         | 52   | 100                          | 15   |
| 25                       | 509                        | +    | 448                         | +    | 40                           | +    |
| 150                      | 5600                       | 2420 | 380                         | 128  | 10                           | 4    |
| <hr/> Cab-0-Sil <hr/>    |                            |      |                             |      |                              |      |
| 0                        | 1120                       | 1230 | 640                         | 160  | 110                          | 20   |
| 25                       | 1620                       | +    | 402                         | +    | 60                           | +    |
| 150                      | 4100                       | 3190 | 426                         | 97   | 15                           | 3    |

+ Not determined

TABLE VI

Solvent Resistance (ASTM D471)  
Mechanical Properties : UCARSIL F<sub>1</sub> Elastomer

| <u>Solvent</u>   | <u>Young's<br/>Modulus,<br/>psi</u> | <u>Tensile<br/>Strength,<br/>psi</u> | <u>Elongation<br/>at Break,<br/>%</u> |
|------------------|-------------------------------------|--------------------------------------|---------------------------------------|
| None             | 1120                                | 640                                  | 110                                   |
| Toluene          | 1566                                | 677                                  | 145                                   |
| Acetone          | 1454                                | 671                                  | 145                                   |
| Reference Fuel B | 1600                                | 603                                  | 120                                   |

TABLE VII

Percent Swelling (ASTM D471)

| <u>Solvent</u>   | <u>Polymer/Filler</u>       |                              |                              |
|------------------|-----------------------------|------------------------------|------------------------------|
|                  | <u>Methylphenyl UCARSIL</u> | <u>UCARSIL F<sub>1</sub></u> | <u>UCARSIL F<sub>1</sub></u> |
|                  | <u>Tullanox 500</u>         | <u>Tullanox 500</u>          | <u>Cab-0-Sil</u>             |
| Methanol         | 1.7                         | 1.5                          | -                            |
| Toluene          | 162                         | 129                          | 98                           |
| Acetone          | 49                          | 71                           | 2.3                          |
| CCl <sub>4</sub> | 146                         | 121                          | -                            |
| Reference Fuel B | 113                         | 100                          | 80                           |

TABLE VIII

Mechanical Properties as a  
Function of Temperature : UCARSIL F<sub>3</sub> Elastomer

| Temp.,<br>°C | Young's<br>Modulus,<br>psi | Tensile<br>Strength,<br>psi | Elongation<br>at Break<br>% |
|--------------|----------------------------|-----------------------------|-----------------------------|
| 290          | 634                        | 16                          | 3                           |
| 260          | 980                        | 26                          | 5                           |
| 200          | 1,020                      | 73                          | 11                          |
| 100          | 1,300                      | 113                         | 60                          |
| 50           | 1,440                      | 126                         | 100                         |
| 25           | 2,245                      | 197                         | 200                         |
| -20          | 259,000                    | 182                         | 1                           |
| -30          | 278,000                    | Broke                       | -                           |

TABLE IX

Mechanical Properties (25°C) after  
Heat Aging in Air at 290°C

| Aging<br>Time,<br>Hrs. | Young's<br>Modulus,<br>psi | Tensile<br>Strength,<br>psi | Elongation<br>at Break,<br>% |
|------------------------|----------------------------|-----------------------------|------------------------------|
| UCARSIL F <sub>2</sub> |                            |                             |                              |
| 0                      | 1420                       | 195                         | 60                           |
| 50                     | 4230                       | 317                         | 20                           |
| 150                    | 7510                       | 505                         | 20                           |
| UCARSIL F <sub>3</sub> |                            |                             |                              |
| 0                      | 2245                       | 197                         | 200                          |
| 50                     | 2590                       | 423                         | 90                           |
| 150                    | 3790                       | 474                         | 30                           |

TABLE X  
Solvent Resistance of UCARSIL F<sub>1</sub>, F<sub>2</sub> and F<sub>3</sub> Polymers (ASTM D471)

| <u>Solvent</u>   | Percent Swelling             |                              |                                                       |
|------------------|------------------------------|------------------------------|-------------------------------------------------------|
|                  | <u>UCARSIL F<sub>1</sub></u> | <u>UCARSIL F<sub>2</sub></u> | <u>UCARSIL F<sub>3</sub></u><br><u>Fluorosilicone</u> |
| Toluene          | 98                           | 63                           | 23                                                    |
| Reference Fuel B | 80                           | 43                           | 20                                                    |
|                  |                              |                              | 21 <sup>a</sup>                                       |

<sup>a</sup> Data from reference 10.